PATENT SPECIFICATION

(11) 1 481 713

1 481 713

(21) Application No. 50942/74 (22) Filed 25 Nov. 1974
(31) Convention Application No. 7343735
(32) Filed 7 Dec. 1973 in
(33) France (FR)
(44) Complete Specification published 3 Aug. 1977
(51) INT CL² B22D 23/08
(52) Index at acceptance
C7X 1

(19)

(54) IMPROVEMENTS IN AND RELATING TO POWDER MANUFACTURE

(71) We, CREUSOT-LOIRE, a French corporate body of 5, rue de Monttessuy, 75007, Paris, France, do hereby declare the invention for which we pray that a patent may be granted to us and the method by which it is to be performed to be particularly described in and by the following statement:—

The present invention relates to the manufacture of a powder of a high-melting point metal in the form of small spherules or globules the size and characteristics of which are controlled.

In the production of a metal powder by the process known as the atomisation of a rotating electrode (REP in the U.S.A.) a cylindrical electrode, or one approximating to a cylinder of revolution, made of the alloy to be powdered is rotated about its axis, this axis being horizontal or vertical.

A point source of heating such as an electric arc melts the free end of the electrode locally (the other end being used for mounting and driving the electrode). The centrifugal force sprays the molten metal in the form of fine droplets which solidify and cool in the course of their free trajectory. In general, the electrode is located in an enclosure having an atmosphere which is neutral in relation to the metal, or under vacuum, in order to avoid a contamination of the powder. This process permits the production of powders of refractory and highly reactive metals such as alloy steels, nickel or cobalt alloys, alloys of titanium, beryllium, niobium, and tungsten.

A known apparatus for carrying out this process comprises a source of heat fixed in the plane perpendicular to the axis of rotation of the rotating electrode. Heating is performed along the axis of rotation of the rotating electrode or with a slight displacement towards the exterior edge of the electrode. Such an arrangement limits the diameter of the rotating electrodes to a few centimetres, since the point heating leads to an excessive hollowing of the electrode by localised fusion. This limitation leads to the following disadvantages:

a) if the device for fixing the rotating electrode is located in the interior of the enclosure, which is the most logical arrangement, the rotating electrode, the length of which is determined by the dimensions of the enclosure, has a relatively low weight because of its small section. The weight of powder obtained in each operation is then very limited (a nickel alloy electrode 4 cm in diameter has a mass of 10 kg per meter);

b) if the device for fixing the rotating electrode is located outside the enclosure, the length of the rotating electrode can be increased. However, straightening must be carried out over the whole length of the electrode, because the sealing joint between the ambient atmosphere and the enclosure will rub on the rotating electrode. Furthermore, the system for driving the electrode must be movable, because it must advance as the rotating electrode is consumed.

The grain size of the powder obtained is connected with the characteristics of the rotating electrode by an equation of the type:

$$d = K\sqrt{\frac{\gamma}{\rho}} \times \frac{1}{n\sqrt{R}}$$

where

d is the diameter of the grains of powder obtained, in microns;

n is the speed of rotation, in revolutions per minute, of the rotating electrode;

R is the radius of the rotating electrode, in centimetres;

γ is the surface tension of the liquid alloy, in ergs/cm²;

ρ is the density of the liquid metal, in g/cm³; and

K is a constant with a value close to 165×10^5.

Thus, to obtain a powder with a given grain size (200μ for a nickel alloy, for example) an electrode with a small diameter requires speeds of rotation of 6,000 to 12,000 rpm, which raises technological problems, the solutions of which are burdensome (sealing joints, bearings).

According to the present invention there is provided apparatus for manufacturing metal

powder by the atomisation of a rotating electrode, having a solid body of revolution which is substantially cylindrical, is made of the material to be atomised and is rotated about its axis, the apparatus comprising an enclosure, means for holding and rotating the electrode about its axis in the enclosure and at least one device for point heating and melting the free end face of the electrode, wherein the point of impact of heat from the or each heating device on the end face of the electrode is movable relative thereto so that substantially the whole of the end face of the electrode when rotating is traversed and heated thereby.

Advantageously, the point of impact of heat from the or each heating device is movable in a direction parallel to the axis of the electrode.

Preferably, when a single source of heat is provided, the source is moved along a radius of the end face of the electrode.

Advantageously, the enclosure that contains the electrode is designed so that the angle that the grains of powder expelled from the rotating electrode make with the wall is between 10° and 60°, preferably close to 45°, in order to avoid crushing the still hot particles against the walls and to avoid their adhesion to the walls. This geometry is made possible without an exaggerated increase in the dimensions of the enclosure by shortening the length of the electrode, a shortening which can be compensated for by an increase in diameter of the electrode.

If the spraying enclosure is filled with a gas which is neutral with respect to the metal to be powdered, the device for point heating may be an electric arc between the electrode to be powdered and a non-consumable electrode or a plasma torch. The non-consumable electrode, or the plasma torch, is attached to a shaft extending parallel to the axis of rotation of the electrode and displaced with respect to this axis by at least half the radius of the rotating electrode. The non-consumable electrode, or the plasma torch, is fixed to the shaft by means of an arm capable of rotational and translational movement in such a way that the electric arc or the plasma can reach the centre of the face of the electrode that is being consumed and, after rotation of the shaft, the edge of this face.

The displacement of the non-consumable electrode or of the plasma torch parallel to the axis of the rotating electrode, designed to compensate for the consumption of the latter, is obtained by the translation of the shaft.

When the spraying enclosure is placed under vacuum, heating may be achieved by an electron gun. This gun may either be fixed to an arm firmly attached to a shaft as described in relation to the electric arc or plasma torch, or, more simply, be fixed. In the latter case, compensation for the consumption of the rotating electrode is achieved by a progressive variation in the focussing of the electron gun; the displacement of the point of impact of the electron beam along a radius of the rotating electrode to obtain a regular consumption of the latter is obtained by deflection of the electron beam. The variation in focussing and the deflection of the electron beam are obtained by electromagnetic means commonly used in electron guns.

In a modification in which heating is obtained, as before, by an electric arc or a plasma torch, two non-consumable electrodes, or two plasma torches, with independent electric feeds, may advantageously be used. These two heating heads may be fixed to the same arm attached to the same shaft, the two heads being on the same radius of the rotating electrode and spaced by a distance which is between one third of the radius and one radius of the rotating electrode and preferably about a half-radius. These heating heads may alternatively be fixed to two independent arms attached to two independent shafts, the arrangement of the shafts and of the arms and the limitation of their movements causing the whole of the frontal surface of the rotating electrode to be swept without the heating heads being able to come into contact.

So that the invention can easily be understood, two embodiments of apparatus according to the invention will now be described, by way of example only, with reference to the accompanying drawing, in which:

Figure 1 is a vertical section through an embodiment of apparatus according to the invention, and

Figure 2 illustrates the arrangement of the rotating electrode and heating means of a modification of the apparatus of Figure 1.

In the embodiment of Figure 1, the electrode 1 to be rotated is fixed to a mandrel 2 through a neck 3 machined in the electrode. The mandrel 2 is connected by a transmission shaft 4 and a perpendicular countershaft 5 to a motor 6 whose speed can be varied from 1000 to 3000 rpm. A bronze disc and a system of graphite contacts 7 enable the shaft 4 to be connected electrically to a direct-current generator (not shown) through electric cable 8. The electrode 1 is brought to a suitable height in enclosure 13 by means of a sleeve 9 with cooled walls and which is slightly displaced with respect to the axis of the enclosure 13 to enable a receiver 12, in which the powder collects, to be placed at the centre of the lower conical part of the enclosure 13.

The enclosure 13 is completely cooled, except in the few places where this is impossible (portholes, flanges, air-tight glands). The upper part 11 of the enclosure contains two portholes 14 and 15, suitably protected against thermal and ultraviolet radiation, which serve for the monitoring of the atomisation process. In its top part there is a cap 16 from which an opening 17 leads to a secondary vacuum pump for evacuating the enclosure before its

filling with a neutral gas through inlet 18. The enclosure is designed so that the angle that the grains of powder expelled from the rotating electrode make with the wall is between 10° and 60°, preferably close to 45°, in order to avoid crushing the still hot particles against the walls and to avoid their adhesion to the walls.

An electric arc is established in operation between the upper face 19 of the rotating electrode and a non-consumable electrode 20. Cooling water and the electric current are supplied by means of a cooled cable 21 which passes through the cap 16, by means of an air-tight gland insulated electrically from the enclosure, before being connected through the electric cable 22 with the direct-current generator. The head of the non-consumable electrode is connected mechanically with, while being insulated electrically from, a horizontal arm 23, itself rigidly attached to a vertical shaft 24 which passes through the upper part 11 of the enclosure by means of an air-tight gland and can be subject to translational and rotational movement for reasons given above by means of two devices 25 and 26 located outside the enclosure.

Such an apparatus with a diameter of about 2.5 m and about 2 m high can atomise rotating electrodes with diameters of 100 to 300 mm and with heights between 20 and 70 cm.

As an example, in the case of a nickel-alloy electrode with a diameter of 120 mm and with a speed of rotation of 300 rpm the following grain-size distribution was obtained:

$<630\ \mu$ — 98.5%
$<500\ \mu$ — 93.3%
$<315\ \mu$ — 21%
$<250\ \mu$ — 14.7%

In a modification of the above described apparatus, two distinct heating heads, e.g. plasma torches or non-consumable electrodes, which never come into contact are used. As shown in Figure 2, the consumable rotating electrode 27 is subjected to the action of two heating heads 28 and 29 which are arranged on two independent arms 30 and 31 each of which is caused to move to and fro in a circular arc. It follows from this that the whole of the end surface of the electrode 27 to be melted is swept by these heads, but the latter cannot come into contact.

There is thus provided apparatus which will permit the production of large batches of powders with an enclosure of reasonable dimensions without burdensome technological complications, by using large-diameter rotating electrodes.

WHAT WE CLAIM IS:—

1. Apparatus for manufacturing metal powder by the atomisation of a rotating electrode, having a solid body of revolution which is substantially cylindrical, is made of the material to be atomised and is rotated about its axis, the apparatus comprising an enclosure, means for holding and rotating the electrode about its axis in the enclosure and at least one device for point heating and melting the free end face of the electrode, wherein the point of impact of heat from the or each heating device on the end face of the electrode is movable relative thereto so that substantially the whole of the end face of the electrode when rotating is traversed and heated thereby.

2. Apparatus according to claim 1, wherein the point of impact of heat from the or each heating device is movable in a direction parallel to the axis of the electrode.

3. Apparatus according to either claim 1 or claim 2, wherein the enclosure is designed such that the angle of impact that the trajectories of the grains of powder expelled from the electrode make with the walls of the enclosure is between 10° and 60°.

4. Apparatus according to any of the preceding claims, comprising a single device for point heating, wherein the point of impact of heat from the heating device on the rotating electrode is movable along a radius of the end face of the electrode.

5. Apparatus according to any of the preceding claims, wherein the heating device is fixed to a shaft extending parallel to the axis of rotation of the electrode, displaced with respect to the said axis of rotation by at least half the radius of the electrode and rotatable so as to sweep the whole of the end face of the electrode when rotating.

6. Apparatus according to any of claims 1 to 4, including means for evacuating the enclosure and wherein the heating device is a fixed electron gun including means for varying the focussing and deflection of the electron beam.

7. Apparatus according to any of claims 1 to 5, wherein the or each heating device is a non-consumable electrode for providing an electric arc or a plasma torch.

8. Apparatus according to claim 7, including two heating devices, with independent electric feeds, fixed to the same arm on the same radius of the electrode and spaced apart by a distance of between one third of the radius and one radius of the electrode.

9. Apparatus according to claim 7, including two heating devices, with independent electric feeds, fixed to two independent arms attached to two independent rotatable shafts, the arrangement of the shafts and the arms and the limitation of their movements causing the whole of the free end surface of the rotat-

ing electrode to be swept without the heating devices being capable of coming into contact.

10. Apparatus for manufacturing metal powder substantially as herein described with reference to the accompanying drawing.

A. A. THORNTON & CO.
Northumberland House,
303—306 High Holborn,
London, W.C.1.

Printed for Her Majesty's Stationery Office, by the Courier Press, Leamington Spa, 1977
Published by The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

BEST AVAILABLE COPY

1481713 COMPLETE SPECIFICATION

1 SHEET *This drawing is a reproduction of the Original on a reduced scale*

FIG:1

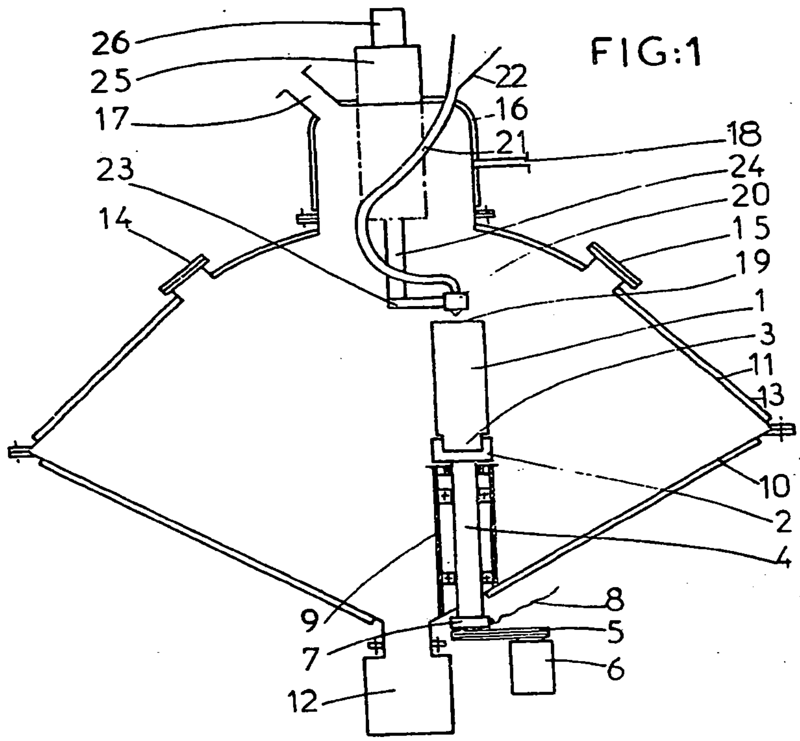

FIG:2